Magnetic-breakdown coupling between orbits of a low-electron-density system

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We have studied by use of the de Haas—van Alphen effect a system of coupled orbits with low charge-carrier density: a single crystal of bromine-intercalated graphite. We have used a new method to determine the effective masses, as well as the de Haas—van Alphen amplitudes at zero temperature for a system with several close de Haas—van Alphen frequencies. We have shown that the Falicov-Stachowiak theory does not give quantitative agreement with our results. Nevertheless, it appears that the Pippard approximation of a network of coupled orbits with Landau numbers of about 15 still remains valid, but the probability of magnetic breakdown should be modified by taking into account the finite size of the magnetic-breakdown area.

I. INTRODUCTION

Several studies, both experimental[1,2] and theoretical,[3–6] have been published on the de Haas—van Alphen (dHvA) effect in systems of coupled orbits with a high number of electrons ($N_0 \sim 10^{23}$ cm^{-3}). Even now the dHvA effect in such systems, with the condition $\omega_c \tau \gg 1$ (ω_c the cyclotron frequency and τ the relaxation time), is not clearly understood.[2] In the semiconductors, where the number of carriers is small, coupling of orbits has been observed neither in the dHvA effect nor in transport effects. We present here the observation of the dHvA effect in a system of interacting orbits with a small number of electrons: an acceptor graphite intercalation compound (GIC).

The acceptor GIC's are obtained by intercalating molecules between graphite layers.[7] During the intercalation process, charge transfer occurs between the graphite host and the intercalated species, which accepts the electrons from the graphite leaving holes delocalized within the graphite layers. This gives a quasi-two-dimensional metal with a carrier density of about 10^{21} cm^{-3} (or 10^{14} cm^{-2}) as determined from both transport and optical measurements.[7] This two-dimensional (2D) system thus has a surface carrier density which is about 100 times larger than that of quantum-Hall-effect devices currently being investigated.[8]

dHvA measurements in acceptor GIC's (Refs. 9—13) show the existence of two types of dHvA frequencies; the basic frequencies and those obtained by linear combinations of the first ones. Such dHvA observations are a clear indication of an orbit-coupling mechanism and make the acceptor GIC's good candidates for the investigation of the dHvA effect in a system of coupled orbits with moderately low carrier density. The band structure currently proposed[7,14] for the acceptor GIC's does not predict such observations and some authors assumed a band folding which takes into account the superlattice of the intercalated molecules.[11]

From dHvA measurements at several temperatures we have been able to determine the effective masses and to obtain the temperature-independent part of the dHvA amplitude as a function of magnetic field. In Sec. II we present the different dHvA-frequency-coupling mechanisms. Section III describes the experimental method and the analysis of the dHvA data; the experimental results are presented in Sec. IV. In Sec. V we interpret the magnetic field dependence of two dHvA amplitudes corrected for the temperature effects by introducing the magnetic-breakdown orbit-coupling mechanism and we fit our results with the Falicov-Stachowiak theory. A discussion of this fit brings us to reconsider the validity of the Pippard approximation of a network of coupled orbits in Sec. VI.

II. dHvA-FREQUENCY COUPLING

A. dHvA effect

The dHvA effect is the quantum oscillation of the magnetic susceptibility of an electron gas as a function of a magnetic field. The frequencies of the oscillations (periodic in $1/H$) are related to the extremal cross sections of the Fermi surface perpendicular to the magnetic field.

Lifchitz and Kosevich have computed the oscillatory part of the magnetic susceptibility of a 3D gas of zero-spin independent electrons[15] [Table I(a)]. However, the acceptor GIC's have a very anisotropic electronic structure which is actually quasi-2D. Table I(b) presents the extension of the Lifshitz-Kosevich theory to the 2D electron gas:[16] the frequency of the oscillation is the ratio of the 2D frequency to the cosine of the angle between the magnetic field and the c axis.

B. Coupling of the dHvA frequencies

Every extremal cross section A_i of the Fermi surface produces in the magnetization an oscillation of frequency F_i and of all of its harmonics, independently from the other areas. As a result, every frequency should appear independently from the others. Experimental results in the GIC's have shown that there are combinations of frequencies, therefore, it is necessary to introduce coupling mechanisms. Different kinds of coupling mechanisms have already been proposed: magnetic breakdown, the effects of the magnetic interactions of Shoenberg[15,17] or the

TABLE I. (a) Oscillatory part of the magnetization according to Lifschitz-Kosevich theory (b) and its extension to a 2D system of electrons. The summation on index i is performed over all the extremal areas of the Fermi surface perpendicular to the magnetic field direction. T_{Di}, m_i, and F_i represent, respectively, the Dingle temperature, effective mass, and dHvA frequency associated with the ith area A_i; the term $\cos[(\pi/2)\gamma_s r(m_i/m_0)]$ was introduced by Dingle to take into account the spin–magnetic-field interaction (γ_s is the gyromagnetic ratio of the electron).

(a)

$$\tilde{M}=\sum_i\sum_{r=1}^{\infty}\frac{D_i^{3D}(H)}{r^{3/2}}\cos\left[\frac{\pi}{2}\gamma_s r\frac{m_i}{m_0}\right]\times\frac{X_{ir}}{\sinh X_{ir}}\exp\left[-X_{ir}\frac{T_{D_i}}{T}\right]\sin\left[2\pi r\left[\frac{F_i}{H}-\gamma\right]\mp\frac{\pi}{4}\right]$$

$$\tilde{M}=\sum_i\sum_{r=1}^{\infty}\frac{D_i^{2D}(H)}{r}\cos\left[\frac{\pi}{2}\gamma_s r\frac{m_i}{m_0}\right]\times\frac{X_{ir}}{\sinh X_{ir}}\exp\left[-X_{ir}\frac{T_{D_i}}{T}\right]\sin\left[2\pi r\left[\frac{F_i}{H}-\gamma\right]\right]$$

$$X_{ir}=2\pi^2 r m_i\frac{k_B T}{|e|\hbar}\frac{1}{H}$$

$$F_i=\frac{\hbar}{2\pi|e|}A_i$$

$$D_i^{3D}(H)=-\frac{F_i e^2}{2\pi^2 m_i}\left[\frac{2|e|H}{\pi\hbar}\right]^{+1/2}\left[\frac{\partial^2 A_i}{\partial k_{\parallel}^2}\right]^{-1/2}$$

$$D_i^{2D}(H)=-\frac{\hbar F_i e^2}{2\pi^2\frac{m_i}{m_0}}$$

stabilization of charge- (or spin-) density waves.[18] One of the differences between the last two, which belong to the family of nonlinear couplings, and the magnetic-breakdown effect, which is magnetic-field-assisted tunneling from one orbit to another, is the temperature dependence. Magnetic breakdown, which is the only temperature-independent effect, generates frequencies with a temperature dependence exactly similar to that of the basic frequencies.

The tunneling of a carrier from one orbit to another by magnetic breakdown can be described in terms of a diffusion process,[19] i.e., Landau-level broadening. As can be seen from the results of the calculation on Bloch electrons in a magnetic field,[3,5,20,21] the shape of the Landau levels can be complex and only the exponential attenuation term in the dHvA amplitude obtained for Lorentzian-Landau levels will be changed.

Falicov and Stachowiak[6] have computed the oscillatory part of the magnetization of a Pippard system of coupled orbits,[4] which gives for uncoupled orbits the Lifchitz-Kosevich formula of Table I. A coupled orbit of N_p magnetic breakdowns and N_q Bragg reflections will contribute to the dHvA spectrum in the following manner. The frequency is equal to a linear combination of the basic frequencies (uncoupled orbits) and the exponential attenuation term in the amplitude will be multiplied by $p^{N_p}q^{N_q}$, with $p^2=\exp(-H_0/H)$ representing the tunneling probability. H_0 is called the magnetic-breakdown field and $q^2=1-p^2$.

The Lifchitz-Kosevich and Falicov-Stachowiak theories for a system of coupled orbits share in common, the intuitive image that the dHvA spectrum is related to the semiclassical trajectories of the wave packets in the presence of a magnetic field. However, Eddy and Stark[2] have shown recently, by dHvA experiments on magnesium single crystals of very high crystalline quality ($\omega_c\tau\gg1$), that the computation of Falicov and Stachowiak is not valid in this limit. This means that when the electrons have the time to propagate along the Pippard network the effects of the crystal lattice invalidate the semiclassical treatments of the magnetic breakdown.

Thus it was interesting to investigate the validity of these ideas for moderate quantum numbers and $\omega_c\tau>1$, which is the case for the acceptor GIC's.

III. EXPERIMENTAL METHOD

Among the acceptor GIC's, we have chosen to study a residual bromine GIC obtained by intercalation of bromine in a natural graphite single crystal by the two-zone method.[22] The sample is one of the few compounds

which is both of good crystalline quality and stable in air and hence can be directly put in the helium bath, allowing a precise knowledge of its temperature.

The samples are small circular slices with a radius R of 2 mm and a depth of 0.2 mm. The axis of the circle is parallel to the c axis of the crystal. Here, we have used the field-modulation method.[23] It can be shown[16] that in this method the presence of a finite resistivity introduces an extra part to the dHvA signal. This part is, for large skin depths ($R/\delta \ll 1$), proportional to $(R/\delta)^2$ and can hence be neglected to the first order in R/δ. In our case we are in the limit of large skin depths, so that any resistive or magnetoresistive effects can be neglected. The resulting signal is detected by a lock-in amplifier and sampled at a constant rate proportional to $1/H$, digitized by a Digital Equipment Corporation MINC 11-03 computer and finally recorded on a floppy disk.

To eliminate the temperature dependence of the data, we evaluated the temperature-dependent term in the Lifchitz-Kosevich formula which depends on the effective masses of the carriers as follows. We first calculated the spectral density, by the discrete fast Fourier transform of the data. The effective masses were then determined by the method described in Sec. I of the Appendix in which we assume that every frequency has a temperature-dependent term $X/\sinh X$ as in Table I, which is true for magnetic-breakdown orbits. The elimination of the temperature dependence was then performed with the computation described in Sec. 2 of the Appendix.

IV. EXPERIMENTAL RESULTS

Figures 1 and 2 represent the rough dHvA data obtained at $T=1.2$ K and $T=7.8$ K and the associated spectral densities obtained by Fourier transform. The values of the corresponding dHvA frequencies are reported in Table II. Here, one should mention the complexity of the dHvA data because of both the number of frequencies and the closeness of the peaks. The results for the effective masses are also represented in Table II. Due to the high spectral density of the dHvA spectra it was not possible to find out the variation of the amplitudes with magnetic field for all the dHvA frequencies, because too close frequencies perturb the computation by generating spurious oscillations. In Fig. 3 and in curve a of Fig. 4, the symbols represent the temperature independent part of the dHvA amplitude variation on a logarithmic scale, versus the inverse of the magnetic field for the peaks at 117 and 78 T. These amplitudes were obtained by the data treatment explained in Sec. 2 of the Appendix. These figures include experimental data at several temperatures. This gives a dispersion of the points which is, however, compatible with the errors in the effective-mass determination, which introduces small spurious temperature dependences. The very small value of the dispersion shows that the temperature dependence of the dHvA amplitude indeed has the form $X/\sinh X$.

V. INTERPRETATION

Now we shall discuss the variation with magnetic field of the temperature-independent part of the dHvA amplitude. The amplitude variation of the 117-T frequency (Fig. 3) is characteristic of a normal orbit, i.e., without any apparent effect of magnetic breakdown in the range of magnetic field we have studied. From these data we obtain, by a least-squares fit, a Dingle temperature of 8 K.

Contrary to that of the 117-T frequency, the amplitude of the 78-T frequency, (curve a of Fig. 4) shows a decrease above a magnetic field of 4 T. Such a decrease cannot be explained by the phenomenological Dingle assumption. In order to explain this variation we assume a coupling mechanism for the dHvA frequencies. Since the results of Fig. 4 curve a show a temperature-independent variation we propose orbit coupling by magnetic breakdown.

In Fig. 4 curve a, the solid line represents the fit of the results by the theory of Falicov and Stachowiak. Four parameters are then necessary: the Dingle temperature T_D of the orbits, the magnetic breakdown field H_0, and the number N_p and N_q of magnetic breakdowns and Bragg reflections along the semiclassical orbit. The best fit is

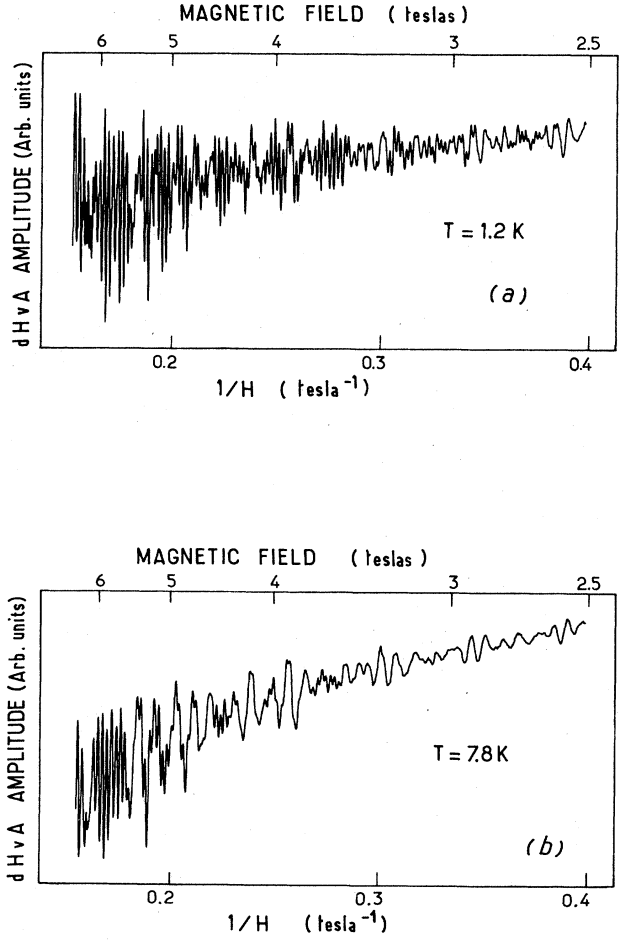
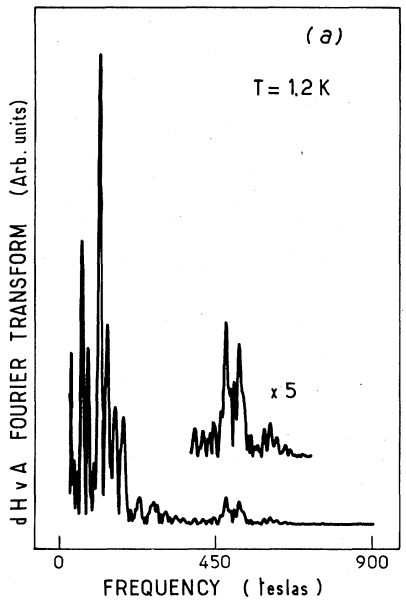


FIG 1. (a) dHvA oscillations, at a temperature of 1.2 K and (b) of 7.8 K, as a function of the magnetic field, in a single crystal of a residual bromine graphite intercalation compound.

obtained for the following values: $T_D = 13.8$ K, $H_0 = 5.4$ T, $N_p = 12$, and $N_q = 46$.

We have made a new computation of the magnetic field dependence of the DHVA amplitude, by correcting now for both temperature and magnetic-breakdown effects. The weight function used (see the Appendix, Sec. 2) is now multiplied by



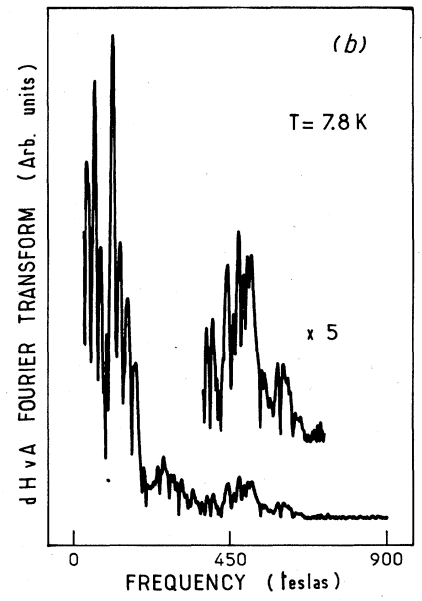


FIG. 2. Discrete Fourier transform of the dHvA oscillations of Fig. 1. Parts (a) and (b) correspond to respective parts (a) and (b) of Fig. 1. The dominant peak has a frequency of 117 T.

TABLE II. dHvA frequencies and effective masses in a dilute graphite-bromine intercalation compound.

Frequencies (T)	Effective masses (in units of the electronic mass)
16±5	0.045±0.006
23±5	0.025±0.003
30±5	0.038±0.006
58±5	0.050±0.0005
76±5	0.065±0.001
84±5	0.110±0.005
95±5	0.090±0.003
117±5	0.091±0.002
126±5	0.096±0.004
150±5	0.070±0.003
176±5	0.099±0.001
190±5	0.110±0.005
236±13	0.15 ±0.02
271±13	0.16 ±0.02
415±13	0.256±0.004
466±13	0.175±0.003
492±13	0.202±0.004
527±13	0.230±0.002
558±13	0.200±0.005
586±13	0.217±0.003
651±13	0.221±0.002

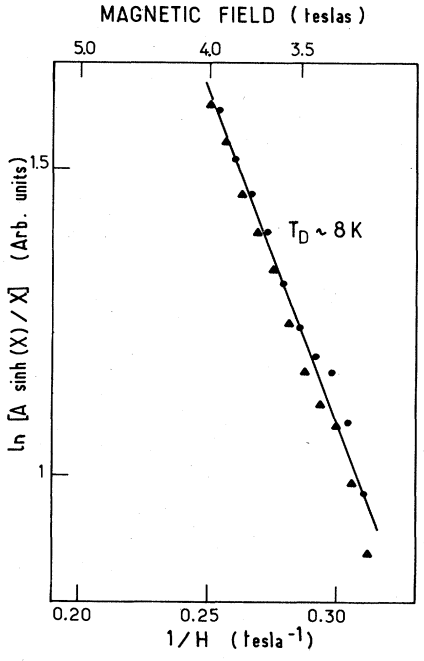


FIG. 3. The variation of the amplitude A of the 117-T dHvA peak, corrected for the temperature effects, as a function of the inverse of the magnetic field. The resulting linear fit gives a Dingle temperature of 8 K.

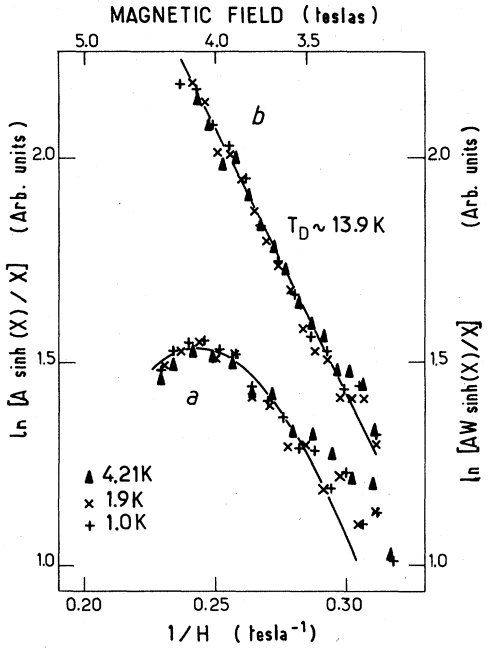


FIG. 4. The variation of the amplitude A of the 78-T dHvA peak as a function of the inverse of the magnetic field. The symbols represent the data obtained by the treatment explained in Sec. 2 of the Appendix. Curve a represents the temperature-independent part of the amplitudes for several temperatures. The solid line is the best fit using the Falicov-Stachowiak theory for the dHvA effect in a system of coupled orbits. In curve b, the amplitudes corrected for both the temperature and the magnetic-breakdown effects are represented. T_D is the Dingle temperature, resulting from the fit represented by the solid line.

$$W=\left\{\left[\exp\left[-\frac{H_0}{H}\right]\right]^{N_p/2} \times\left[1-\exp\left[-\frac{H_0}{H}\right]\right]^{N_q/2}\right\}^{-1}$$

with the parameters of the best fit, which takes into account the magnetic-breakdown effects. The result of the computation is represented by the symbols on curve b of Fig. 4, for several temperatures. The distribution of points can be well fitted by a line. The least-squares fit (solid line) then gives a Dingle temperature $T_D \sim 13.9$ K. The computation could not be done for fields above 4.16 T because perturbations are introduced by the presence of adjacent peaks. From the value of the Dingle temperature, 13.9 K, we obtain a relaxation time $\tau=8.7\times10^{-14}$ sec; we have then $\omega_c\tau=1.56$ for $H=6.6$ T which was the highest field used in our experiments. This means that the quantum condition is not fulfilled.

VI. DISCUSSION

Our results show that the functional variation predicted by the Falicov-Stachowiak theory for a system of coupled orbits is in agreement with our results. However, we found very high values for two of the parameters of the fit, N_p and N_q. These give a very complex semiclassical orbit. Indeed the observation of such an orbit would be accompanied by the observation of numerous smaller and simpler orbits, predicted by Pippard's[4] model of the 2D network of orbits in real space on which the Falicov-Stachowiak theory is based. We have not observed such an orbit.

For GIC's we have previously explained the dHvA experimental results with a network of coupled orbits in reciprocal space.[11] This network is obtained by adding to the band structure of s charged interacting carbon layers[14] the effect of the in-plane order of the intercalate layer. Thus we obtain a network of intersecting semiclassical orbits with many magnetic breakdowns and Bragg reflections. Therefore, we observe abnormal behavior in a system which presents a Pippard orbit network. We attribute this anomaly to the spatial extent of the semiclassical orbit.

In our experiments the quantum numbers of the Landau levels are between 10 and 100, compared to a few thousands in metals. If R_C and R_Q are, respectively, the radius of the semiclassical and quantum orbits[5,15] then the relative extension of the wave packet given by $(R_C-R_Q)/R_C$ cannot be neglected and the intersection zones of the semiclassical orbits of the 2D-orbit network are not more localized but are extended spatially. This means that the semiclassical approximation of the Pippard 2D network is not valid for systems with a low number of electrons, i.e., low quantum numbers of the Landau levels.

We can estimate the spatial extent of the orbit if we remember that the ratio of the classical and quantum areas is $(n+\gamma)/n$, where n is the quantum number of the Landau level and $0<\gamma<1$. For a circular orbit with $n\sim15$ and $\gamma\sim0.5$ we have $(R_C-R_Q)/R_C\sim0.03$. The qualitative agreement of our experiments, as seen in Fig. 4, with the Falicov-Stachowiak theory, shows that when the junctions of the 2D Pippard-network orbits are extended spatially, Pippard's treatment remains a valid approximation, but the amplitudes of the magnetic-breakdown probability should then be modified. The large N_p and N_q values that we have found from our experimental results represent this effect. An alternative approach was developed by Hofstadter[20] and Claro and Wannier[21] who calculate the Landau levels in a tight-binding model in very high magnetic fields but not the resulting dHvA effect in such a situation: they found an absence of periodicity in $1/H$. This makes the comparison with dHvA experiments very difficult.

VII. CONCLUSION

We have studied by the de Haas—van Alphen effect, a system of coupled orbits with low charge-carrier density: a single crystal of bromine-intercalated graphite. We have

used a new method to determine the effective masses as well as the dHvA amplitude at zero temperature for a system with several close dHvA frequencies. The coupling mechanism is the magnetic breakdown. Our results show the functional variation predicted by the theory of the dHvA effect in a system of coupled orbits that was proposed by Falicov and Stachowiak. However, we cannot give the usual meaning to the values obtained for the number of magnetic breakdowns and Bragg reflections in the semiclassical orbit (the adjustable parameters in the expression). The Pippard 2D orbit network can still be used for a system with low quantum numbers, but the magnetic breakdown probability should be computed in accordance with the spatial extent of the magnetic-breakdown zone.

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APPENDIX

We describe here the methods for computing the effective masses and for eliminating the temperature dependence in the dHvA amplitude from a complex dHvA spectrum. Both computations use the discrete Fourier transform in a fast Fourier transform (FFT) algorithm.

1. Effective-mass computation

As can be seen in Table I, the effect of the temperature is to multiply each amplitude by a temperature-dependent factor. This factor will be called the trial function $F_i(H)_{\{m_i,T\}}$ for the ith dHvA frequency. In this function the magnetic field H acts as the variable while m_i and T are parameters. In order to compute the value of the parameter m_i we multiply the μth dHvA data set, obtained at T_μ, by

$$F_i(H)_{\{m_i,T_\nu\}}/F_i(H)_{\{m_i,T_\mu\}} ,$$

where m_i is equal to a trial value. FFT is then applied to the νth data and to the transformed μth data. The best trial value of the parameter m_i is the one for which the ith amplitudes obtained for the transformed μth and νth spectra are equal.

The validity of this numerical treatment requires the fulfillment of the three following conditions.

(1) The dHvA data at the different temperatures have to be sampled identically during the acquisition.

(2) The signal-to-noise ratio has to be high and the temperature fluctuations have to be small.

(3) The temperature difference $|T_\mu - T_\nu| = \Delta T$ should be sufficiently large. It can be shown[16] for instance that it is impossible to determine an effective mass of 0.01 times the free-electron mass if $\Delta T < 10$ K.

2. Determination of the temperature-independent part in the dHvA spectra

We want to determine the temperature-independent part of the ith dHvA-frequency amplitude; we apply the FFT operation to the dHvA data recorded at the temperature T and weighted by the function $1/F_i(H)_{\{m_i,T\}}$, where m_i is the effective-mass parameter and $F_i(H)_{\{m_i,T\}}$ is the trial function defined in Sec. 1 above.

The succession of FFT computations on overlapping field domains $[1/H_1, 1/H_2]$ such that $\Delta(1/H) = 1/H_1 - 1/H_2$ is constant allows us to construct the numerical function $A_1(1/H)$, with

$$1/H = (1/H_1 + 1/H_2)/2 ,$$

equal to the value of the module of the FFT for the ith frequency in the dHvA spectrum, computed on this domain. So we approximate $A_i(1/H)$ by the temperature-independent term of the ith amplitude in the dHvA data.

The validity of this approximation is based on the following: starting from the definition of the discrete Fourier transform[24] it is easy[16] to show that $A_i(1/H)$ is equal to the sum of two parts. The first part alone is the sum on all the dHvA amplitudes, averaged on $[H_1, H_2]$, of the temperature-independent term multiplied by a slowly varying sinusoidal function of frequency smaller than or equal to the uncertainty of the FFT computations. The second part is made of terms crossed with the other amplitudes of the dHvA spectrum. For the ith dHvA peak sufficiently far (a few full widths at half maximum) from its neighbors, the contribution of the crossed terms is negligible and the field variation can be well approximated by the dependence in

$$1/H = (1/H_1 + 1/H_2)/2$$

on the average values sampled on $[1/H_1, 1/H_2]$. One should then make a compromise on the value of $\Delta(1/H)$: large values give negligible crossed terms, while small ones give a better equivalence between an average value and the value at a given point. The error made in the determination of the m_i parameter adds a temperature-dependent contribution to the above computed term, proportional to the relative error $\delta m/m$.

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